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Microstructural Characterization and Some Mechanical Behaviour of Low Manganese Austempered Ferritic Ductile Iron

A. A. Abioye[1*], P. O. Atanda[2], O. P. Abioye[1], S. A. Afolalu[1] and J. O. Dirisu[1]

[1]Department of Mechanical Engineering, Covenant University, P. M. B. 1023, Ota, Ogun State. Nigeria.
[2]Department of Materials Science and Engineering, Obafemi Awolowo University, Ile-Ife, Osun State. Nigeria.

Abstract

This work studied the microstructural characterization and mechanical behavior of low manganese Austempered Ductile Iron (ADI), with a view to improve the properties of iron and to increase the areas of applications. Three sets of ductile iron of specified composition were machined from Y-blocks to tensile and hardness pieces. The samples were preheated at 350 ^{0}C for 1hr and austenitised at 900 ^{0}C for 1hr in salt bath furnace. The three sets of samples were immediately austempered in the austempering salt bath furnace at uniform austempering temperatures of 300 ^{0}C, 350 ^{0}C and 400 ^{0}C for 90, 120 and 150 minutes; each sample for each temperature window. All sets were prepared for metallographic examination; tensile and hardness tests were carried out. The results showed that maximum hardness, tensile and yield strength were obtained at austempering temperature of 350 ^{0}C and at 150 minutes. At 300 ^{0}C and 350 ^{0}C, it was noticed that the hardness and strength increase with austempering time. The optimum tensile strength was 1300 MPa at 350 ^{0}C after austempering for 150 minutes. In conclusion the austempering operation has a significant effect on the mechanical and microstructural properties of ADI.

Keywords: Ausferrite, Spherodial-Graphite, Acicular phase

INTRODUCTION

Examining the effects of heat treatment on microstructure evolution is important because the microstructure of a metal affects the strength and physical behavior of the metal. There are different types of microstructures for different alloys. The types of phases present, the volume fraction of the phases, the grain size, and grain shape determine the properties, which in turn govern the appropriate application of the alloy. Austempered ductile iron (ADI) has a microstructure containing spherodial graphite embedded in a matrix which is in general a mixture of phases called ausferrite [1]. There are different phases present in ADI but the most desirable phases are acicular ferrite and high retained carbon austenite. The structure of the matrix surrounding the free graphite nodules is engineered, depending on the applied isothermal heat treatment schedule and conditions, to produce a specific austempered 'steel like' microstructure consisting of acicular, carbide-free ferrite (ausferrite) with controlled amounts of retained austenite (an fcc ductile phase). The austempering process also produces a material which has mechanical properties comparable with those of forged steels [2].

ADI has other technical and commercial advantages which include: lower density than forged steel, better vibration damping, improved lubrication properties, lower raw-material costs, substantial energy savings during manufacture for particular components, nearly twice as strong as pearlitic ductile iron, ADI retains high elongation and toughness [3-5]. These properties are achieved by heat treatment of an alloyed ductile iron using an austempering process. An optimum combination of high carbon austenite and acicular ferrite confers excellent mechanical properties of such cast iron. This in turn allows a wide range of applications with ADI competing favourably against forged steel and aluminum alloys in terms of mechanical properties, manufacturing cost, physical properties and weight savings.

Austempering heat treatment is a general method of producing ausferritic matrix structure in ductile iron. The heat treatment is essentially a two stage operation. The first stage is austenitising at a temperature range of 815 – 920˚C for between 1 to 4 hours. The specific austenitising temperature selected is related to the subsequent austempering temperature and the grade of ADI required. Once austenitising temperature and time are selected, close control over them is essential. This is followed by rapid quenching in a salt bath where the castings are held isothermally at selected austempering temperature. Austempering temperatures are in range 230 – 450˚C according to the properties required in the castings. At higher austempering temperatures (upper bainitic range), ferrite structure nucleates and grows into austenite [6-8]. Lower austempering temperatures, produce finer and greater volume fractions of ferrite, and higher yield strength. The nature of ausferrite microstructure depends not only on austempering time but also temperature. Higher austempering temperatures produce a coarser ferrite but a lower volume fraction of ferrite. This is accompanied by lower yield strength [9, 10]. The purpose of this research was to produce austempered ductile iron from ferritic ductile iron, and determine the effect of the phases present in its microstructure on the mechanical properties of the produced austempered ductile iron.

MATERIAL AND METHODS

Three sets of test samples were machined from the leg part of the Y-blocks of ductile iron, whose chemical composition (wt %) is shown in Table 1. The samples were machined to test pieces for tensile and hardness properties. The tensile and hardness test samples were machined as per ASTM standard E-8 and E 602-91. The samples were preheated to 350ºC to avoid cracking, distortion on subsequent austenitization. Thus, the temperature difference between the austenitic region and the preheated regime was brought closer so as to avoid these heat treatment defects. The austenitization was carried out in the austenitizing salt bath furnace containing a mixture of $BaCl_2$ and NaCl, in ratio 3:2 at 900ºC and was held at constant time period of 1 hour to dissolve the carbon in the austenite. The set of samples were immediately transferred (to avoid pearlite formation) to the austempering salt bath of $NaNO_3$ and KNO_2, in ratio 1:1 at uniform austempering temperatures of 300ºC, 350ºC and 400ºC and were held for 90, 120 and 150 minutes respectively. Finally it was air cooled to room temperature.

The set of samples for each austempering temperature and time conditions were prepared for micro-examination in accordance with ASTM E407-07. The Metallography process: sample sectioning, mounting, grinding, polishing and etching were carried out on the samples. The microstructure was examined under optical microscope and scanning electron microscope after etching with 2% NITAL. Tensile testing of all the specimens was conducted as per ASTM Standard E-8. Set of identical test sample for each austempering temperature and time conditions and as cast ductile iron was tested at room temperature with a crosshead speed of 1 mm min-1 using a computerized Instron 3369 electromechanical testing machine. Load- displacement plots were obtained on an X-Y recorder and the ultimate tensile strength and yield strength values were calculated from this load-displacement diagram. The set samples for each austempering temperature and time conditions and as cast ductile iron sample were subjected to Brinell hardness test using the Houndsfield extensometer in compression mode. A 10 mm indentor made of a hardened steel ball was mounted in a suitable holder and will be forced into the prepared surface of the specimens polished to 600 microns using a dwell time of 15 seconds. The diameter of the impression left by the ball was measured using the Brinell calibrated hand lens and the corresponding Brinell hardness number was determined. The hardness of each test pieces was taken at five different locations and the average was determined as hardness value. The Brinell hardness number (BHN) was evaluated according to equation (1.0):

$$BHN = 2F/(\pi D\,(D - \sqrt{(D_2 - d_2)})) \quad (1)$$

Where

F = Imposed load (kg)

D = Diameter of the spherical indenter (mm)

d = Diameter of the resulting indentation (mm)

X-ray diffraction analysis was performed to estimate the accicular ferrite and the high carbon retained austenite. It was done using a monochromatic copper K_α radiation at 40 KV and 100 mA. A Rigaku rotating head anode diffactrometer was used to scan the angular 2θ range of 42 - 46˚ at a scan speed of 0.25˚ per minute and 72 - 92˚ at a scan speed of 1˚ per min. Thereafter the profiles were analyzed using PANalytical X' Pert Pro powder difractometer in θ - θ configuration with an X' Celerator detector and variable divergence and fixed receiving slits with Fe filtered Co-Kα radiation (λ= 1.789Å). The phases present were identified using X' Pert Highscore plus software. Also, the peak positions and integrated intensities of (111), (220) and (311) planes of austenite and (110) and (211) planes of BCC ferrite were obtained from the profile.

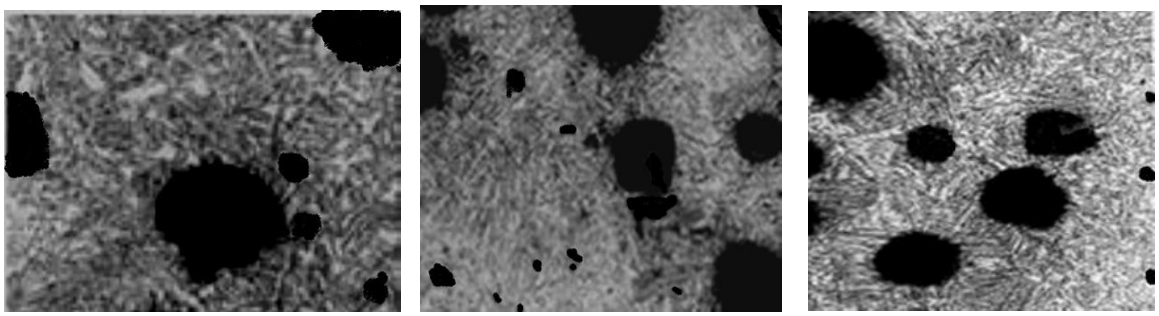
RESULTS AND DISCUSSION

The optical micrographs of the produced ADI at different austempering temperatures and time were as shown in Figure 1 to Figure 3. The micrographs were for the produced ADI at different austempering temperatures of 300ºC, 350ºC and 400ºC and austempering time of 90, 120 and 150 minutes respectively. The microstructures consisted of spheroidal graphite in a matrix of acicular ferrite and stabilized austenite (called ausferrite). The structures of samples austempered at 350ºC and 400ºC were characterized by coarse ausferrite platelets with more coarse structures at 400ºC while the structure of samples austempered at 300ºC was characterized by fine needles of ausferrite. Significant coarsening of ausferrite was observed as the austempering time increases. Figures 4 to 6 were the SEM micrographs of samples austempered at the above austempering parameters. The structures showing a matrix of acicular ferrite and stabilized austenite (called ausferrite). Like optical micrograph the structure of the samples austempered at 400ºC and 350ºC were characterized by coarse ausferrite platelets while the structure of samples at 300ºC was characterized by fine needles of ausferrite and significant coarsening of ausferrite was observed as the austempering time increases.

Table 1: Chemical composition of investigated Austempered Ductile Iron (wt%)

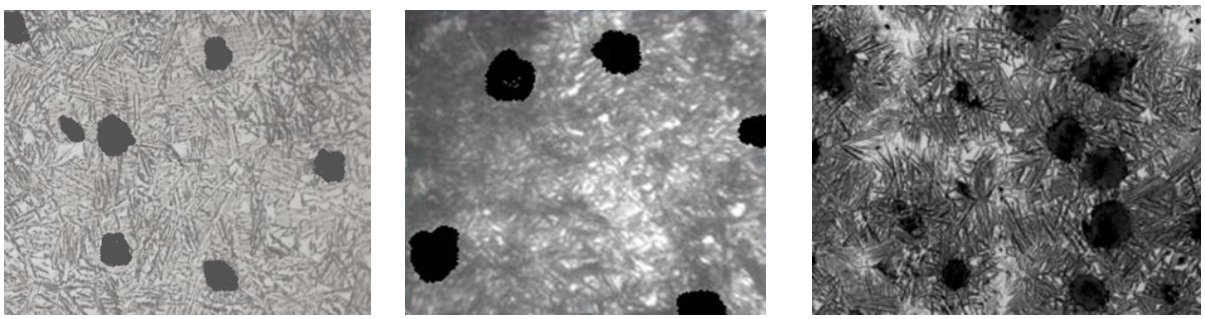
Element	C	Si	Mn	P	S	Cu	Cr	Ni	Mg	Ti	Al	Fe
% Composition	3.620	2.550	0.400	0.017	0.018	0.017	0.048	0.012	0.080	0.072	0.015	93.151

The XRD patterns of produced ADI were as presented in Figures 7 - 9 also confirmed that the produced ADI predominantly consist of ausferrite structure. From the pattern, the peak values were predominantly austenite (γ) and ferrite (α) phases. The austenite (γ) phase was observed on (111) plane at 2Θ of 42.9879^0, (002) plane at 2Θ of 50.0813^0 and (220) plane at 2Θ of 73.6085^0 for the produced ADI samples at different austempering parameters. The ferrite (α) phase was observed on (110) plane at 2Θ of 44.5690^0, (200) plane at 2Θ of 64.9270^0 and (211) plane at 2Θ of 82.2477^0 for the produced ADI samples at different austempering parameters. The mechanical results of produced ADI samples and the standard samples were presented in graphs form in Figure 10 (a) – (c).



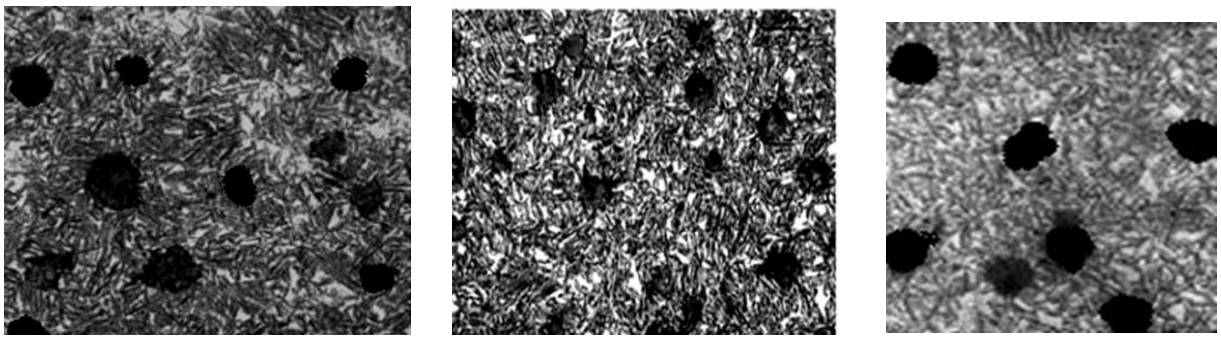
(a) **(b)** **(c)**

Figure 1: Optical micrographs of ADI at 300˚C (a) 90mins. (b) 120mins (c) 150 mins (X 400)



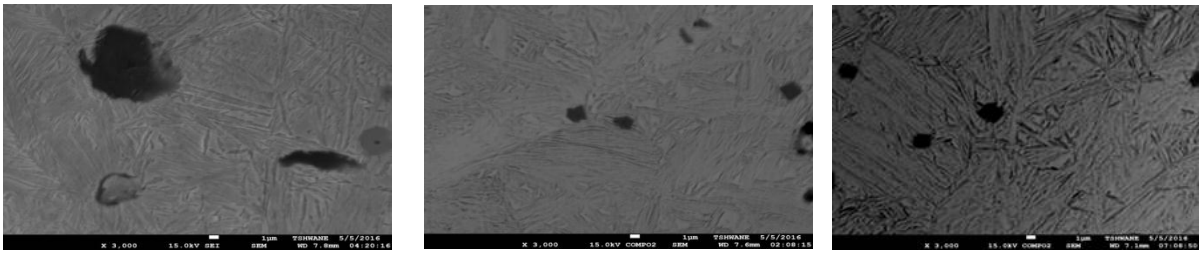
(a) **(b)** **(c)**

Figure 2: Optical micrographs of ADI at 350˚C (a) 90mins. (b) 120mins. (c) 150mins. (X 400)



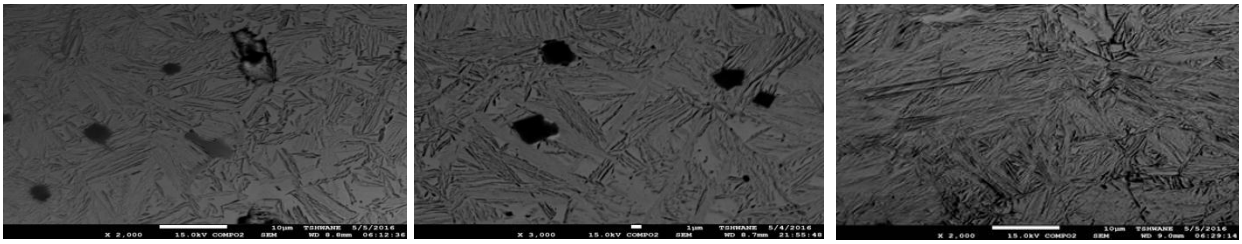
(a) **(b)** **(c)**

Figure 3: Optical micrographs of ADI at 400˚C (a) 90mins. (b) 120mins (c) 150mins. (X 400)



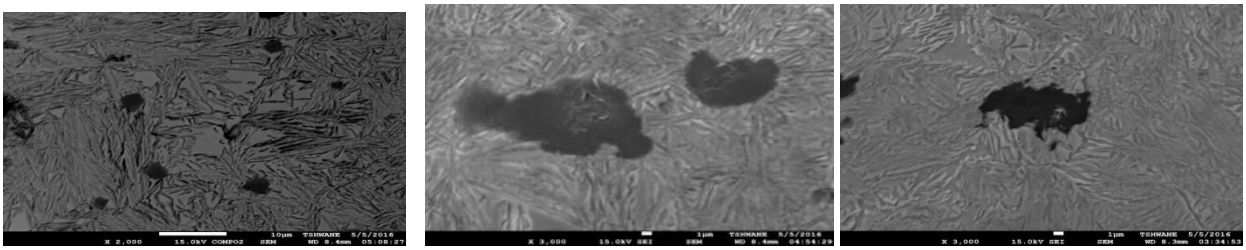
(a) **(b)** **(c)**

Figure 4: SEM micrographs of ADI at 300 ˚ C (a) 90mins. (b) 120mins. (c) 150mins.



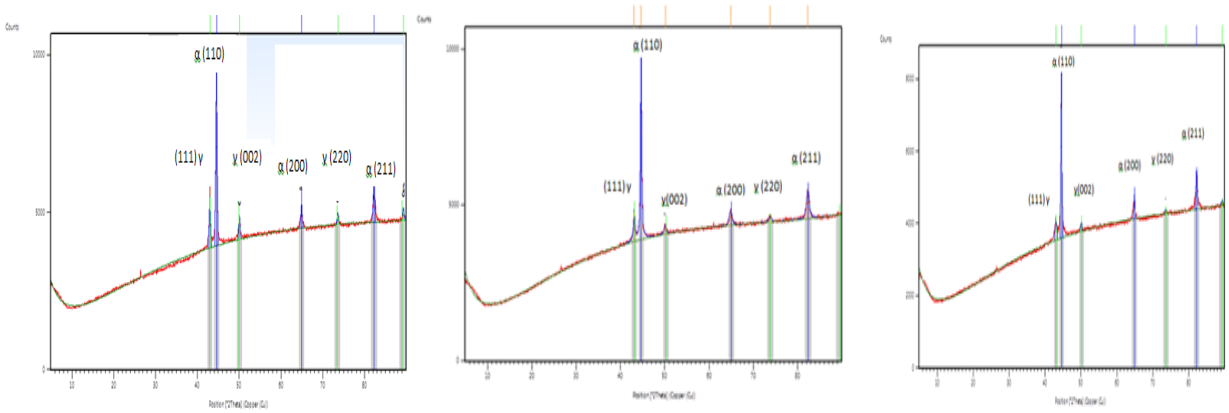
(a) **(b)** **(c)**

Figure 5: SEM micrographs of ADI at 350 ˚ C (a) 90mins. (b) 120mins. (c) 150mins.



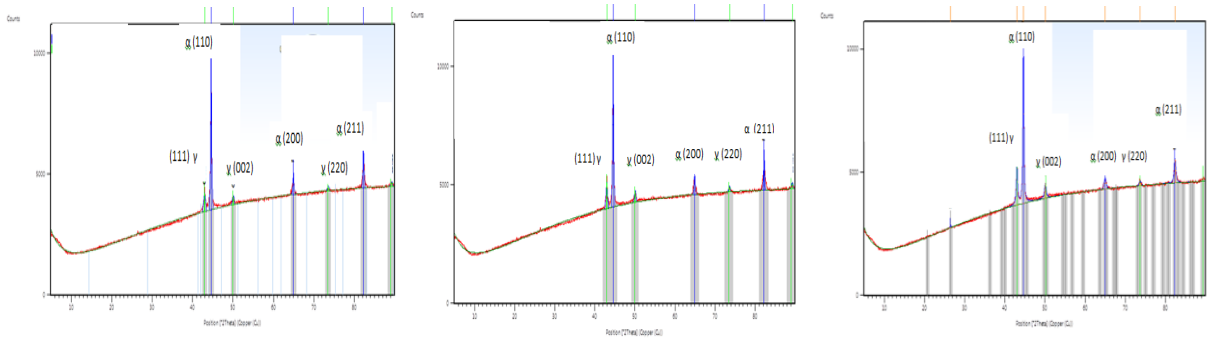
(a) **(b)** **(c)**

Figure 6: SEM micrographs of ADI at 400 ˚ C (a) 90mins. (b) 120mins. (c) 150mins.



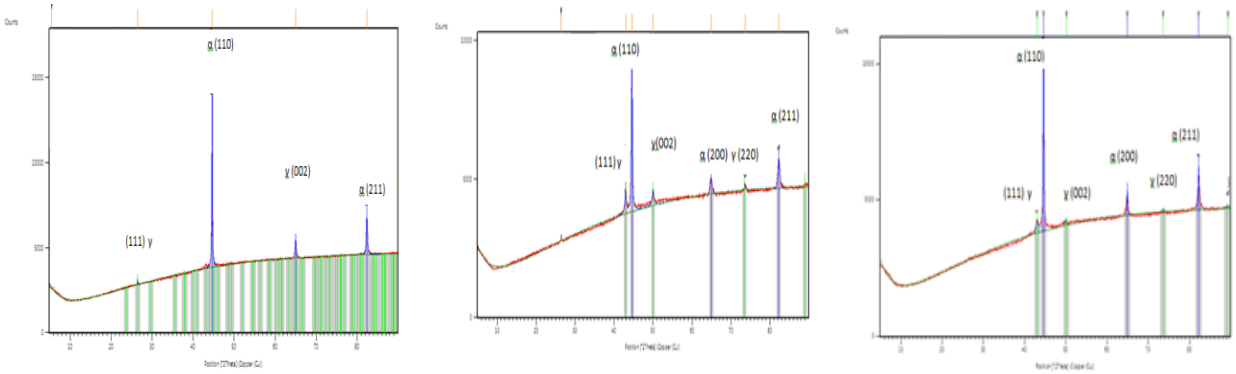
(a) **(b)** **(c)**

Figure 7: XRD of ADI at 300 ˚ C (a) 90 mins. (b) 120 mins (c) 150 mins



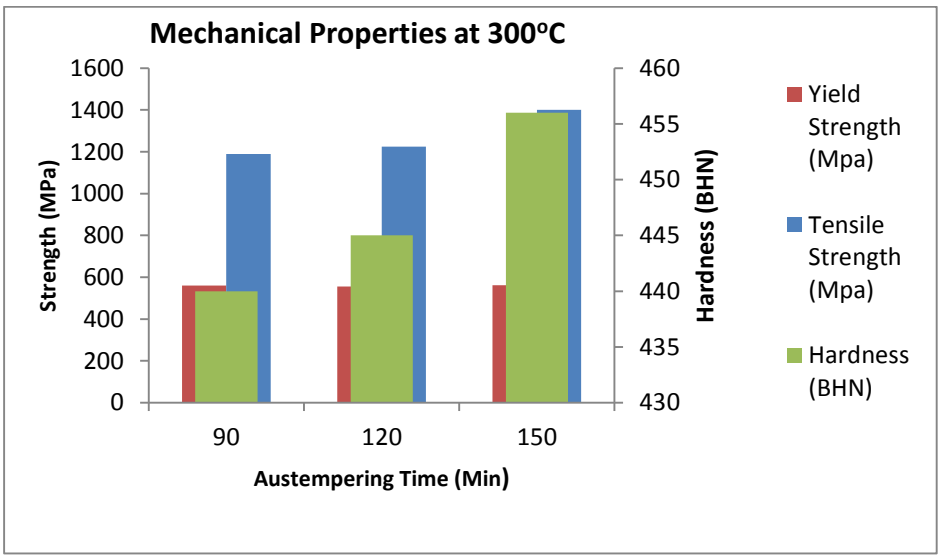
(a) **(b)** **(c)**

Figure 8: XRD of ADI at 350˚C (a) 90mins. (b) 120mins. (c) 150mins.

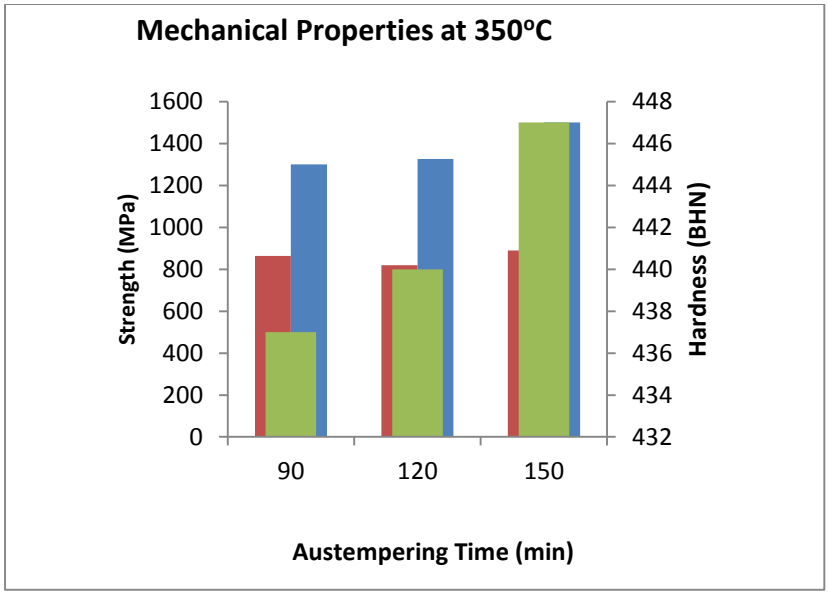


(a) **(b)** **(c)**

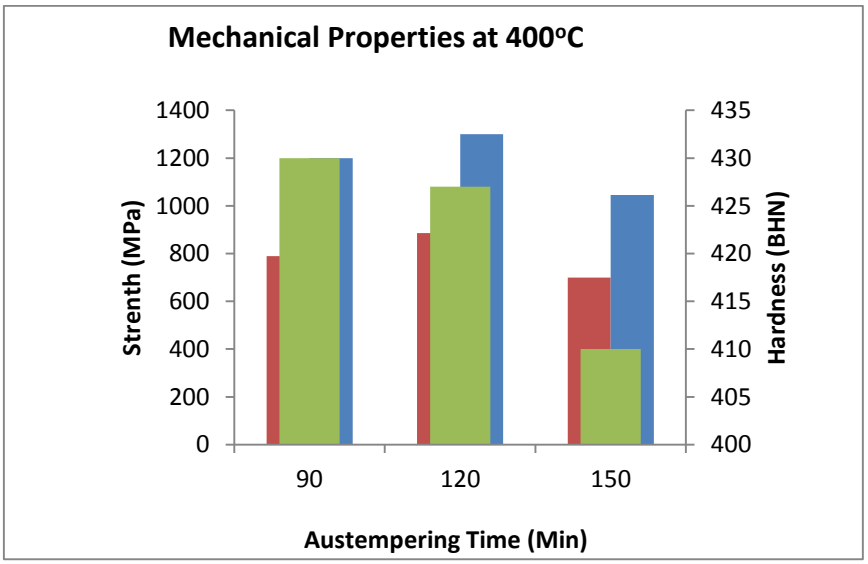
Figure 9: XRD of ADI at 400˚C (a) 90mins. (b) 120mins. (c) 150mins.



(a)



(b)



(c)

Figure 10 (a) Mechanical Properties of produced ADI at 300˚C at different Austempering Time **(b)** Mechanical Properties of produced ADI at 350˚C at different Austempering Time **(c)** Mechanical Properties of produced ADI at 400˚C at different Austempering Time

At isothermal temperature treatment of 300˚C, growth rate of ferritic needles are high and as the rate of carbon diffusion is relatively low, this results in high carbon content in acicular ferrite. The specimen austempered at 300˚C (Figure 1a –c and Figure 4 a - c) showed a typical lower ausferrite microstructure with acicular appearance of ferrite. In specimen austempered at 300˚C for 90 minutes, only small quantities of retained austenite were present, this was because little carbon was rejected from acicular ferrite into the residual austenite. This was clearly seen in the micrographs shown in Figures 1 (a) and 4 (a). At longer austempering time, the carbon enrichment is sufficient to stabilize the austenite even after air-cooling having effect on the retained austenite by increasing it, this could be shown in Figures 1 (b,c) and Figure 4 (b,c) micrographs. The samples treated at austempering temperature of 350˚C for 90, 120 and 150 minutes, all have upper ausferrite as the predominant microstructures. The upper ausferrite consisted of fine ferrite plates as could be seen in Figures 2 (a) - (c) and Figure 5 (a) – (c). The acicular appearance of ferrite in the matrix of retained austenite was also present in the microstructure after austempering at 350˚C. It was observed that this temperature, the time and

temperature of isothermal transformation during the austempering treatment have a marked influence on the relative amount of retained austenite. At this temperature there was a noticeable increase in the amount of retained austenite when compared with samples austempered at 300˚C, there seemed to be balance between the retained austenite and acicular ferrite. The acicular structure at this temperature were 'feathery', coarser, more plate-like morphology of upper ausferrite as described by Spanos, 1994 in his work as compared to lower ausferrite morphology at 300˚C [11]. As the austempering treatment increases from 90 to 150 minutes the acicular structure became more feathery and the amount of retained austenite increases. The samples treated at austempering temperature of 400˚C for 90, 120 and 150 minutes, all have upper ausferrite as the predominant microstructure Figures 3(a) - (c) and Figure 6(a)-(c) . The structures are more feathery and coarser than that of those austempered at 350˚C. These more coarse feathery structures were pronounced using SEM as shown in Figures 4- 6.

At 300°C and 350°C it was noticed that the hardness, yield strength and tensile strength increases with increase in austempering time and also the these mechanical properties were higher at 350°C when compared to 300°C as shown in Figure 10 (a) and (b). At 400°C as shown in Figure 10 (c) the hardness decreases with increase in austempering time. It could be deduced that the ADI sample has the best properties at austempering time of 120 minutes, the hardness properties at this temperature decreases as the austempering time increases. It could be seen that there was a sharp decrease in the mechanical properties of ADI between the austempering time of 120 and 150 minutes this might be because of the formation of carbide. From the mechanical results obtained, the optimum mechanical properties were observed at austempering temperature of 350°C with the best mechanical properties at austempering time of 150 minutes as shown in Figure 10 (b).

CONCLUSION

Austempering temperature has a significant effect on the phase transformation of the Ductile Iron during austempering heat treatment. The higher the austempering temperature the more coarse the structure of the ausferrite present in the microstructure. This could be deduced in the discussion of the result above. The austempering time was also a major factor that influenced the phase transformation of the produced ADI, this could be deduced from the coarsening of the ausferrite as the austempering temperature increased from 90 minutes to 150 minutes. The result of the XRD confirmed that the basic phases present in the samples were predominantly austenite (γ) and ferrite (α) phases, which proved that the produced ADI is a microstructure containing spherodial graphite embedded in a matrix which is in general a mixture of phases called ausferrite.

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